Basic hands-on gamma calibration for low activity environmental levels

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Presentation outline

- Detector characteristics
- Commonly used types of detectors
- Calibration of detectors
  - Energy
  - Resolution
  - Efficiency
- Efficiency calibration of HPGe detectors
  - Experimental calibration
    - Reference data used for calibration
    - Commonly used calibration standards and approaches
    - Preparation of volume standards
    - Calibration curves
    - Commonly encountered problems
  - Geometry, self-attenuation and coincidence summing corrections
  - Combined experimental and numerical calibration
- Demonstration of GammaVision calibration routine

Basic characteristics of gamma-ray spectrometric systems

Environmental gamma-ray spectrometry nowadays relies on low-background high resolution HPGe detectors, with performance characterized by

- efficiency
- resolution
- peak/Compton ratio

Important characteristic:
the system's background,
which can be decreased using

- low activity detector components
- passive shield (lead + copper, tin, plexiglass)
- active shield (anti-cosmic, anti-Compton)

…but more information comes with a detector on delivery

Specifications

- Detector model & s/n
- Cryostat model
- PA model
- Nominal volume
- Resolution @ 122 & 1332 keV
- Peak/Compton ratio
- Endcap dimensions

Physical characteristics of crystal

Electrical characteristics

Resolution and relative efficiency

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DETECTOR SPECIFICATION AND PERFORMANCE DATA

Specifications

Detector Model GCW20025 **Serial number** b 02139

Cryostat Model 7915-30-ULB-CD

Preamplifier Model 2001C

The purchase specifications and therefore the warranted performance of this detector are as follows :

Nominal volume 800 cc

Resolution 2.5 keV (FWHM) at 1.33 MeV

____ keV (FWTM) at 1.33 MeV

____ keV (FWHM) at 122 keV

____ keV (FWTM) at ____

Peak/Comton ____:1 Cryostat well diameter 25 Well depth 60 mm

Cryostat description or Drawing Number if special U-style Integral, type 7915-30-ULB-CD

Physical Characteristics

Geometry Coaxial one open end, open end facing window

Diameter 102 mm

Length 102 mm Crystal well depth 56 mm

Distance from window (outside) 10 mm Crystal well diameter 33 mm

Electrical Characteristics

Depletion voltage (+)5000 Vdc

Recommended bias voltage Vdc (+)5500 Vdc

Leakage current at recommended bias 0.1 nA

Preamplifier test point voltage at recommended voltage -1.1 Vdc

Resolution and Efficiency

With amp time constant of 6 µs

Isotope	^{57}Co	^{60}Co			
Energy (keV)	122	1332			
FWHM (keV)	1.55	2.31			
FWTM (keV)		4.32			
Peak/Compton					
Rel. Efficiency					

- Test are performed following IEEE standard test ANSI/IEEE std325-1996
- Standard Canberra electronics used - See Germanium detector manual Section 7

Tested by : Date : June 17, 2002

Approved by : Date : June 17, 2002

CANBERRA Semiconductor is an ISO 9000 certified company

…and even more is available upon request from the supplier*

The data provided by the supplier are necessary, but not sufficient, for modeling the detector.

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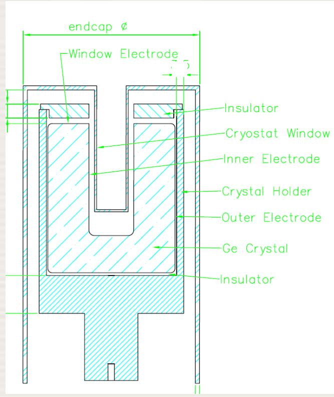

Ge crystal details

Detector chamber details (materials and dimensions)

Exact plans are confidential, certain parameters are even not known exactly to the producer, but by combining experiment and modeling, a set of effective detector parameters can be defined.

*ask for it when you buy the detector, otherwise you may have to pay for it

…and finally the off-the-shelf solution

Have your detector characterized by the producer
(for an additional cost, but may finally save money)

Laboratory Sourceless
Calibration Software

(LabSOCS CANBERRA)

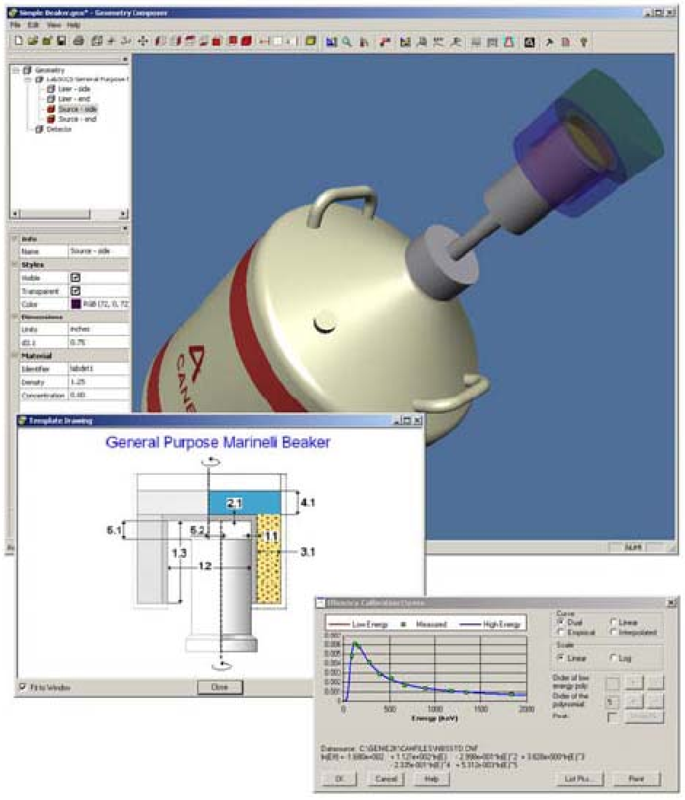

Figure 1
LabSOCS Calibration Software is launched, used for data entry, and then generates the Efficiency Calibration Curve.

- Eliminates the cost of purchasing, tracking, and disposing of radioactive standards
  - No radioactive sources needed for accurate efficiency calibrations in the laboratory
- Calibrations valid from 10 keV to 7000 keV

Not yet developed for well detectors!

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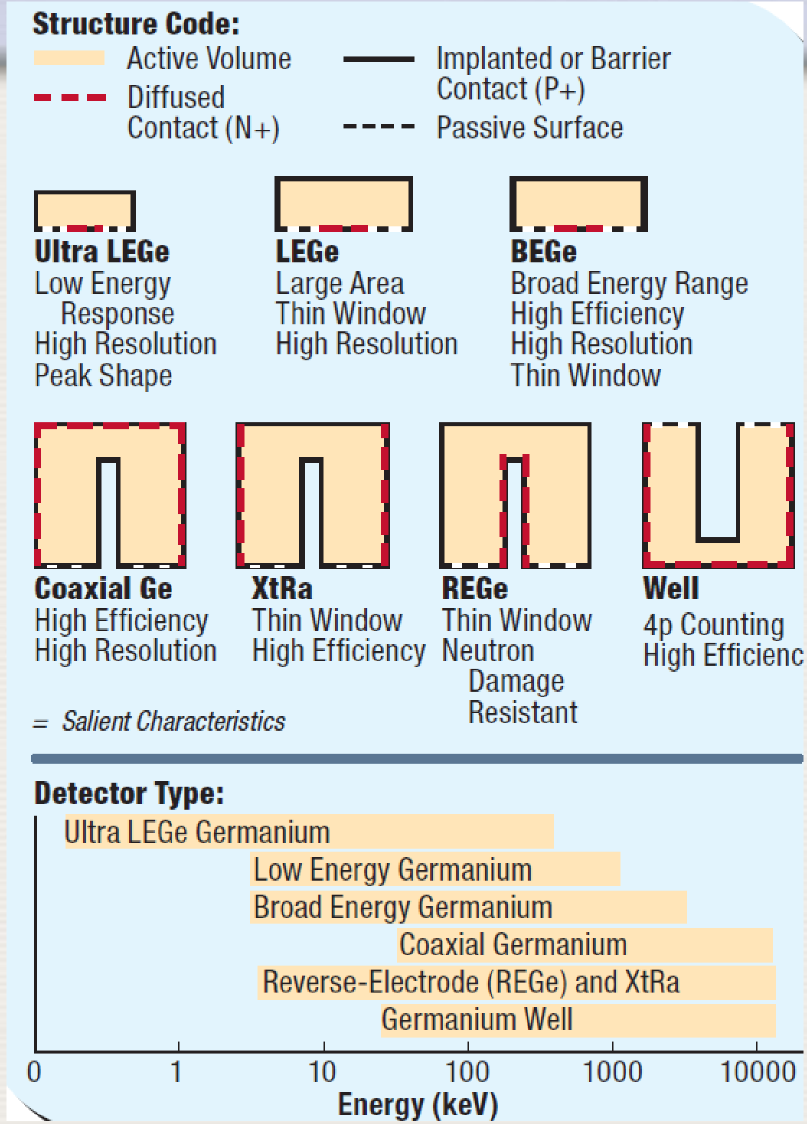
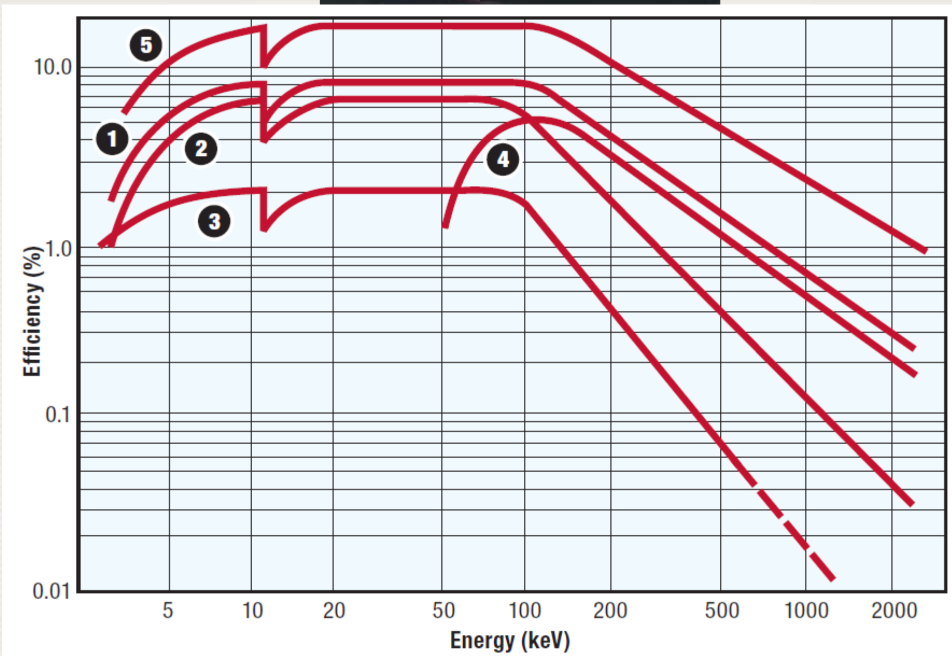
Commonly used types of Ge detectors





Typical absolute efficiency curves for various Ge detectors with 2.5 cm source to end-cap spacing

1. REGe, 15% Relative Efficiency XtRa, 15% Relative Efficiency
2. LEGe, 10 cm^2 x 15 mm thick
3. LEGe, 200 mm^2 x 10 mm thick
4. Coaxial Ge, 10% Relative Efficiency
5. BEGe, 5000 mm^2 x 30 mm thick

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Common types of semiconductor detectors used for environmental applications

Type of Radiation			Detector Type
Charged Particle	γ-Ray	X-Ray	
	X	X	NaI(Tl) Scintillation Detectors – Standard sizes to 3 x 3 in. (7.6 x 7.6 cm). Consult factory for sizes not represented, including annuli for Ge Compton Suppression Systems.
X			PIPS Charged Particle Detectors – Complete line of rugged detectors for alpha and/or beta charged particle analysis.
	X	X	Ultra-Low Energy Ge Detectors – Spectroscopy from 0.3 to 300 keV.
	X	X	Low Energy Ge Detectors – Low Energy Photon Spectrometers for the energy range of 3 to 500 keV.
	X	X	Broad Energy Ge Detectors – High efficiency and resolution from 3 keV to 3 MeV.
	X		Coaxial Ge Detectors – General γ-ray Spectroscopy from 50 keV to 10 MeV.
	X	X	XtRa Detectors – γ-ray Spectroscopy from 3 keV to 10 MeV.
	X	X	Reverse-Electrode Ge Detectors – Radiation Damage Resistant – Spectroscopy from 3 keV to 10 MeV.
	X		Ge Well Detectors – Near 4π counting efficiency for small samples.
		X	Si(Li) Detectors – For X-ray Spectroscopy – 30 keV and below.
		X	Array Detectors – For EXAFS and related applications.
		X	X-PIPS Detectors – Peltier cooled, Silicon x-ray detectors – spectroscopy from 1 to 30 keV.

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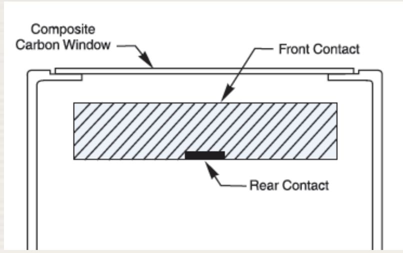
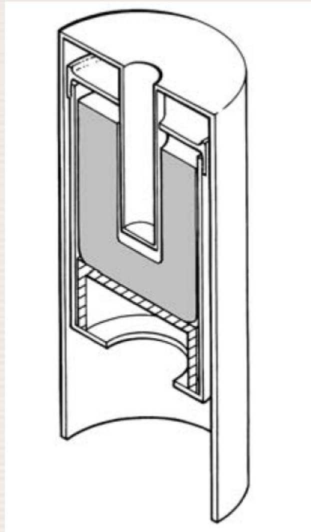
How to chose your detector?

Most environmental gamma spectrometrists aim primarily to minimize the MDA (minimum detectable activity)

- Detection efficiency
- Background
- Resolution





Other issues to consider:

Energy range of interest
Efficiency vs. energy
Typical sample size
……

Some common-sense tips:

- Large ultra-low background well detectors will be good for low single-line radionuclides (e.g. Cs-137), but require the user to master coincidence corrections for other applications

- Broad energy Ge detectors are excellent for normal environmental levels

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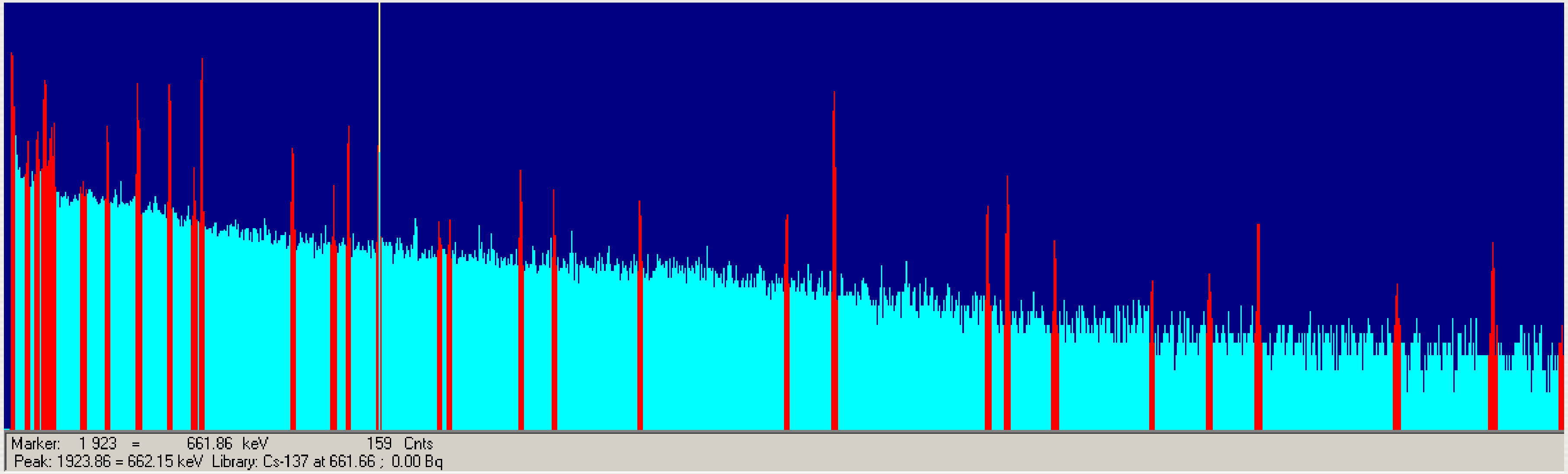
Important: choice of adequate shielding!!!

Why do we need calibration?

To interpret a spectrum in terms of

- energy (identify radionuclides) → energy calibration
- activity (quantify radionuclides) → efficiency calibration

To carry out automated spectrum analysis → peak width calibration



Sediment spectrum, 200ks 200% well, Pb+Cu+anti-cosmics shielding (GammaVision)

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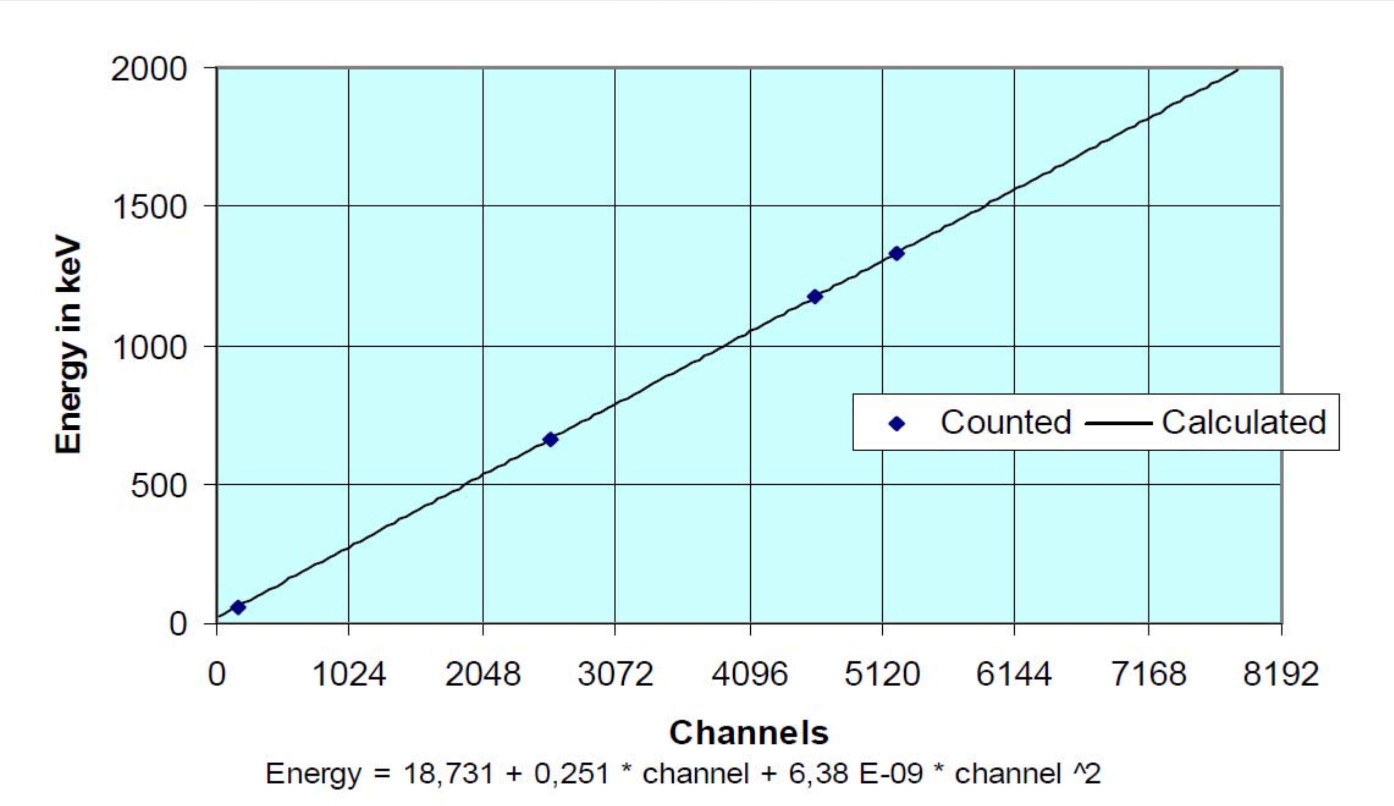
Energy calibration

Energy = A + B * CH + C * CH^2

Energy = A + B * CH   sufficient in general with nowadays electronics for environmental applications



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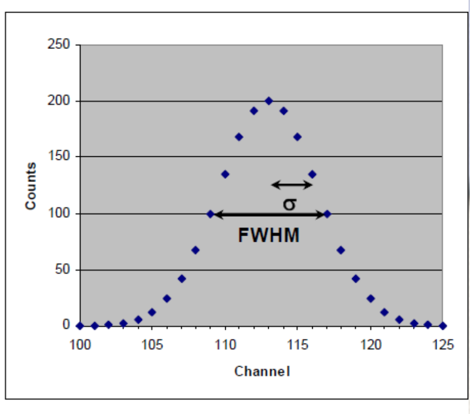
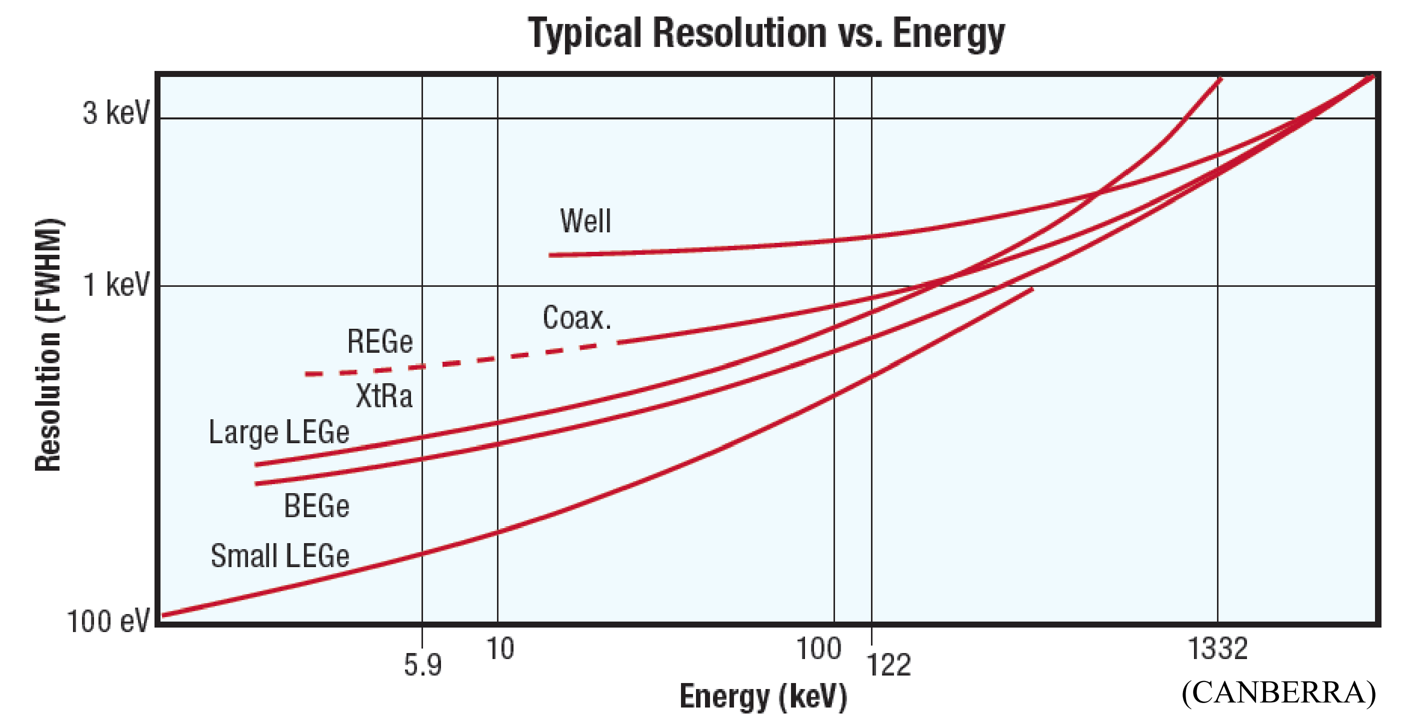
Peak width calibration

$$FWHM = a * E^{1/2} + b * E + c$$



$FWHM = 2.35 * \sigma$



Other functions may apply depending on the energy range and taking into account the peak shape

Efficiency calibration: which efficiency is used in common applications?

Relative efficiency

a general performance measure relating the efficiency of detection of the 1332 keV Co-60 gamma ray for a point source placed centrally 25cm above the detector to that of the standard 3" x 3" sodium iodide scintillation detector

Absolute full energy peak efficiency (FEPE)

the ratio of the number of events when the complete energy E was deposited in the detector to the number of photons of energy E emitted from the source

Absolute total efficiency

relates the number of gamma-rays emitted by the source to the number of counts detected anywhere in the spectrum. This takes into account the full-energy peak and all incomplete absorptions represented by the Compton continuum

Intrinsic efficiency (full energy peak or total)

relates the counts in the spectrum to the number of gamma-rays incident on the detector. This efficiency is a basic parameter of the detector

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Efficiency calibration

- The detection efficiency depends on
  - the energy of the incident gamma ray
  - the detector crystal (intrinsic)
  - the materials surrounding the crystal
  - the source/sample - detector geometry
  - attenuation in the source matrix
  - …

- Can be determined experimentally measuring calibration standards with known activity.

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Reference decay data (NUCLEONICA)

International Committee on Radionuclide Metrology (ICRM)
President: Dr. Pierino De Felice
    http://physics.nist.gov/Divisions/Div846/ICRM/
Recommendations for the development of a consistent set of decay data

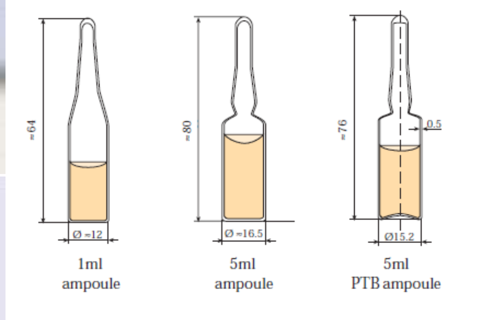
Decay Data Evaluation Project
    http://www.nucleide.org/DDEP.htm
Careful evaluation
Periodic updates

NUCLEIDE database and software (evolved from Tables des Radionucleides) – LNHB
    http://www.nucleide.org/

BIPM Monographie – 5
    http://www.nucleide.org/Publications/monographie_bipm-5.htm

BNL, ENSDF – larger number of nuclides, less dedicated evaluations
Nuclear Data Sheets
    http://www.nndc.bnl.gov/ensdf/

Efficiency calibration: standardized solutions



Mixture	Energy range [keV]	Nuclides	Mixture recommended by	Notes
NG1	80-1836	Ba-133,Co-57,Ce-139,Sr-85, Cs-137,Mn-54,Zn-65,Y-88	PTB	The half lives of the component radio-nuclides are relatively long (shortest is 65 days, Sr-85). At close source-detector distances, summation effects become important.
NG2	88-1836	Cd-109,Co-57,Ce-139,Hg-203, Sn-113,Sr-85,Cs-137,Co-60,Y-88	NIST	The shortest half life is 47 days (Hg-203). The Hg should be precipitated as a sulphide if the solution is dried, to avoid loss of the radioactivity. Summation effects are less important than for NG1.
NG3	60-1836	Am-241, Cd-109,Co-57,Ce-139, Hg-203,Sn-113,Sr-85,Cs-137, Co-60,Y-88	NIST (modified)	As NG2, but extends the calibration down to 60keV.

Efficiency calibration: standardized solutions

NG4	46-136	Pb-210,Am-241,Cd-109,Co-57	PTB	Intended for low energy calibration only (46-136keV).
NG5	88-1836	Cd-109,Co-57,Ce-139,Cr-51, Sn-113,Sr-85,Cs-137,Co-60,Y-88	NIST (modified)	The shortest half life is 28 days (Cr-51). Preparation of solid standards is easier than for NG2 and NG3, as the Cr-51 replaces the Hg-203.
NG6	60-1836	Am-241,Cd-109,Co-57,Ce-139, Cr-51,Sn-113,Sr-85,Cs-137, Co-60,Y-88	NIST (modified)	As NG5, but extends the calibration down to 60keV.
new NG7	60-1836	Am-241,Cd-109,Co-57,Ce-139, Hg-203,Sn-113,Sr-85,Cs-137, Mn-54,Co-60,Zn-65,Y-88	NIST (modified)	As NG3, but with Mn-54 and Zn-65 for high accuracy calibration.

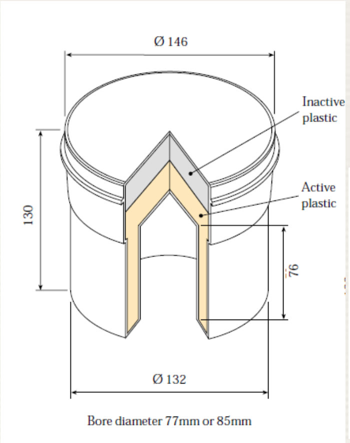
Efficiency calibration: standardized solutions

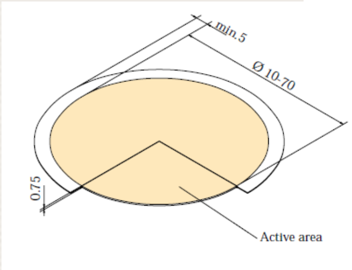
- Attention to the uncertainties specified by the supplier! These will enter into the uncertainty budget of the results. Choose solutions with <2% uncertainty, slightly higher (3-4%) acceptable for some radionuclides (e.g. Pb-210)

- Not all solutions can be mixed! Because of the different chemical forms precipitates can form and you will have an inhomogeneous secondary standard! E.g. NG4 (Pb-210) should not be mixed into any of the other solutions.

- Do not use Reference Materials for calibration. Their use is limited to internal quality control and proficiency tests.

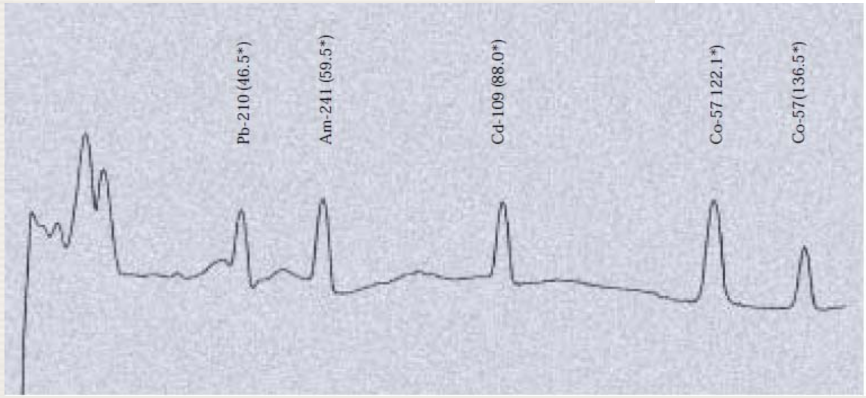
Geometry reference sources – commercially made to order

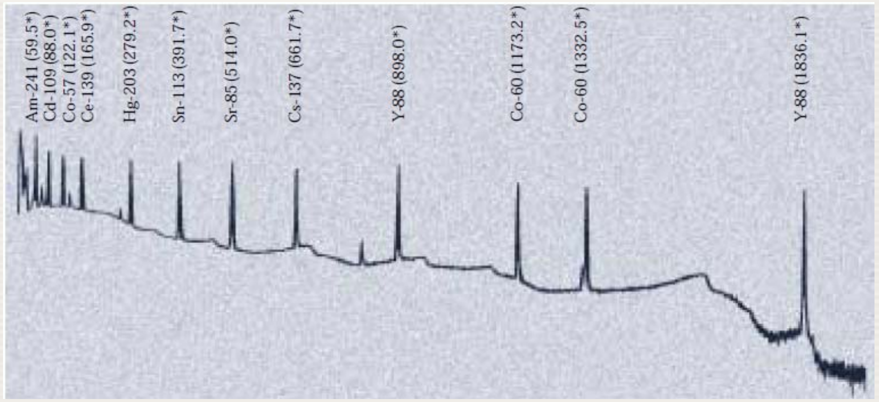




Mixed or single radionuclide solutions are incorporated homogeneously into a plastic resin which is then poured into the container and allowed to set. The active resin is normally covered by a layer of inactive resin. A lid is fixed to the container to make a sealed, solid, source.

(QSA www.nuclitec.de)





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Geometry reference sources – commercially made to order

- You can order your own geometries, preferred densities and radionuclide mixes
- Corrections will need to be applied when matrix is important

Recommended radionuclide mixtures (composition see pages 99 - 101):

NG1 ☐ NG2 ☐ NG3 ☐ NG4 ☐ NG5 ☐ NG6 ☐

Total activity: ____________

Other radionuclides/activities: ____________

For beakers/bottles/containers:

Active volume: ____________ ml

Density: ____________ g/cm^3

For simulated filters:

Active diameter: ____________ mm

Overall diameter: ____________ mm

Date required by: ____________

Number of sources required: ____________

Container material: ____________

Please sketch the container.

The plastic resins used cannot be used with some containers. If the container is not suitable, would you like us to offer an alternative that matches closely the dimensions of your container?

Yes/No

Thank you. Please send 3 empty containers to your local office for testing, marking your name and address clearly on the container.

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Geometry reference sources – commercially made to order

- No source preparation needed - saves time and resources
- No dilution of solutions needed - calibration is directly traceable to national standards
- No dispensing of strong acids for dilution is needed - source is safer to handle
- Sealed source - no risk of contaminating sensitive equipment
- Stable source - will not deteriorate over time
- Source checked for homogeneity - consistent, accurate, results year after year
- Matches closely the samples to be measured - can be prepared in your own container (see page 91) with a wide range of densities from gas equivalent (0.02g/cm^3) to cement (3g/cm^3)

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Geometry reference sources – secondary standards made in the lab

Preparation and storage of mixed gamma stock solution

Procedure presented separately

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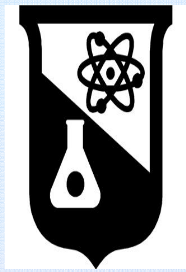
Preparation of mixed gamma standard in sediment

I. Levy, J. Bartocci

1) *Take a quantity of sediment, previously counted, adequate for the geometry of interest. Weigh the sediment.*

2) *Weigh the mixed gamma solution spike and dilute in appropriate acid solution to amount sufficient to cover sediment.*



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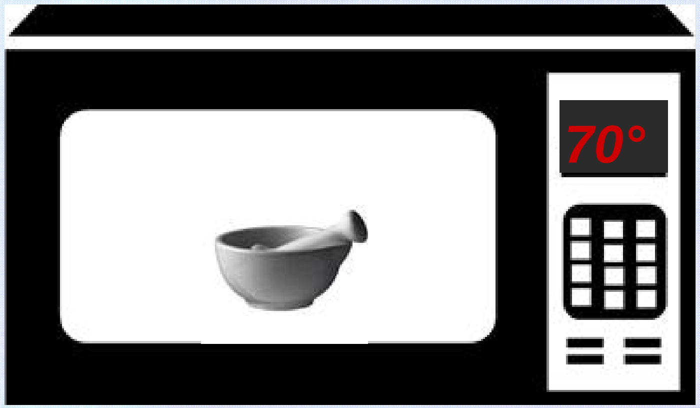
3) Put the sediment into a mortar and pour gently the weighted mixed gamma solution.

Rinse three times the beaker with the appropriate acidic solution and add to sediment.

4) Put the mortar with sediment and mixed gamma solution in an oven for 12 hours.



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5) Put the mortar into a dessicator for cooling.

6) Gently homogenize the sediment with the pestle.

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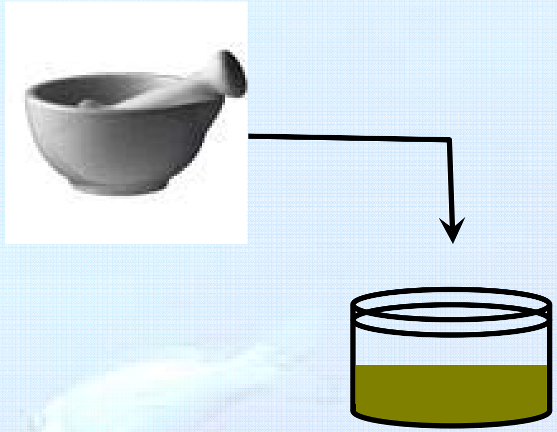
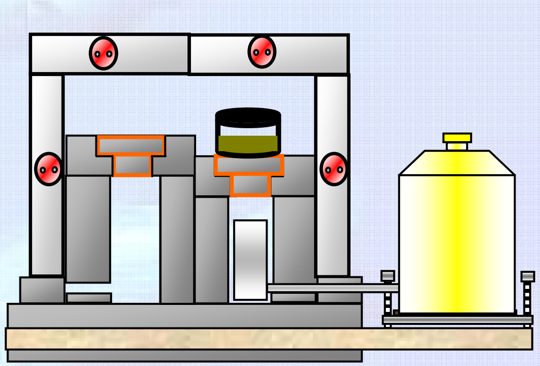
7) Pour the prepared sediment into the measurement container.



Tare the capped container, then weigh with the sediment added in the desired geometry to determine the exact weight of sediment used.

8) Count the secondary volume standard.



The container cap should be taped to avoid contamination! Mind GLP and safety rules when preparing and handling standards. Beware to contaminate detectors!

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Efficiency calibration: experimental

$$\varepsilon(E) = \frac{A_n^i}{\Lambda^i \cdot Y(E) \cdot t} \cdot K_1$$

ε(E) – FEPE for a gamma ray of energy E

A_n^i – net area under the full energy peak of radionuclide i

Λ^i – reference activity of radionuclide emitting gamma-ray of energy E

Y(E) – emission probability of radionuclide at energy E

t – spectrum collection time (live time)

K_1 – decay-correction factor reference date – counting date

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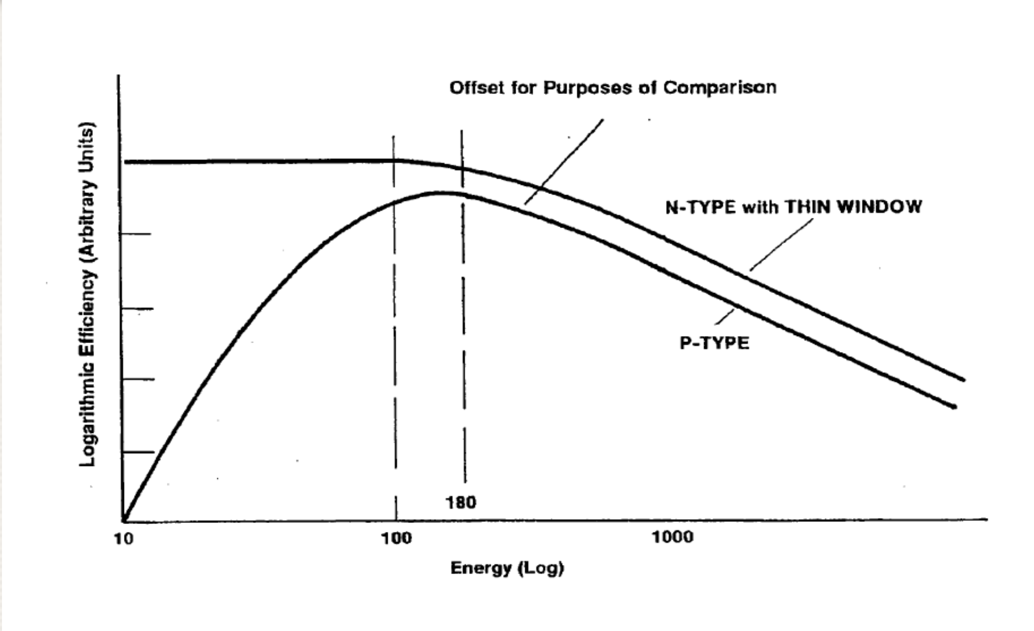
Efficiency calibration (FEPE)



Various functions and combinations of functions can be used to describe the energy dependence of efficiency.

Example

$$\ln \varepsilon(E) = a_0 + a_1 \cdot \ln(E) + a_2 \cdot \ln^2(E)$$

where ε - efficiency, E - energy

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Efficiency calibration: experimental

- Ideally: calibration standard identical in all aspects (geometry, matrix, radionuclide composition) to sample. In this case a direct calibration can be made for the radionuclides of interest.

- Many different radionuclides can be present in the sample, therefore efficiencies may be required at energies other than those of the gamma rays emitted by the calibration standard. Calibration curves have then to be determined based on the experimental efficiencies calculated for the gamma-rays emitted by the calibration standard.

- All commercial software include several options for the type of fit used to describe the efficiency-energy dependency (linear, quadratic, polynomial). You can also use your own routines to fit the experimentally obtained efficiencies.

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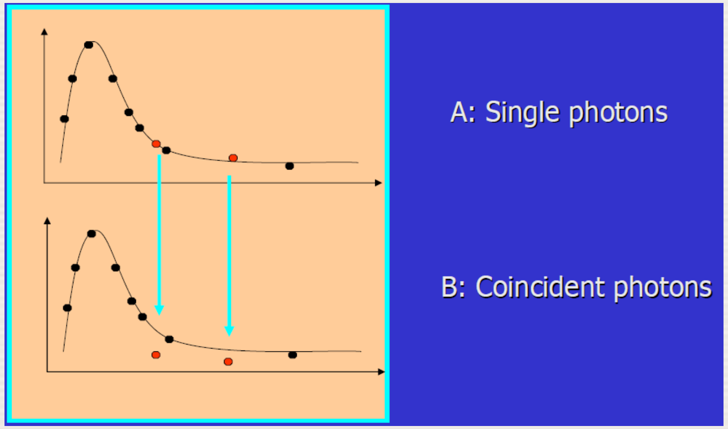
Commonly encountered problems

- Sample and calibration standards not identical (matrix, density)
- The numerous counting geometries and matrices usually encountered in an environmental lab require many calibration standards: costly procurement, storage and disposal
- Radionuclides with coincident gamma-ray emissions in standard and sample



Combined experimental and numerical approach

Corrections

- Geometry: different geometry sample – standard
- Self-attenuation: important for low energy gamma rays when the sample and the standard have different composition (matrix effect) or density (differently compacted)
- Coincidence summing: important for radionuclide with cascade lines in close counting geometries, particularly in well detectors

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Combined experimental and numerical approach

Corrections can be calculated based on

- analytical models
- Monte-Carlo simulations

Monte-Carlo simulations can also be used to calculate the efficiency curve and to optimize counting geometries

There are other approaches: simulations using point source measurements, efficiency transfer, using particular radionuclides for calibration

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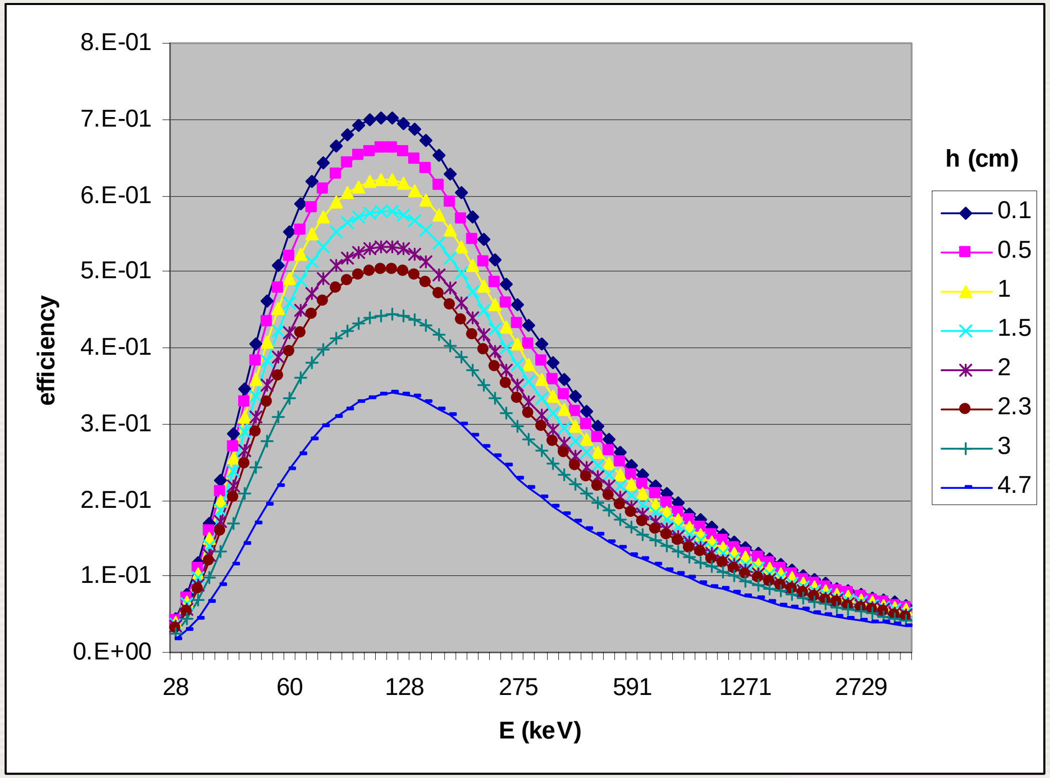
Geometry: to be respected!



Well detector, 150% relative efficiency
Geometry: cylindrical tube r = 1cm, h = 0.1 – 4.7cm
Water

(Monte-Carlo calculations, GESPECOR)

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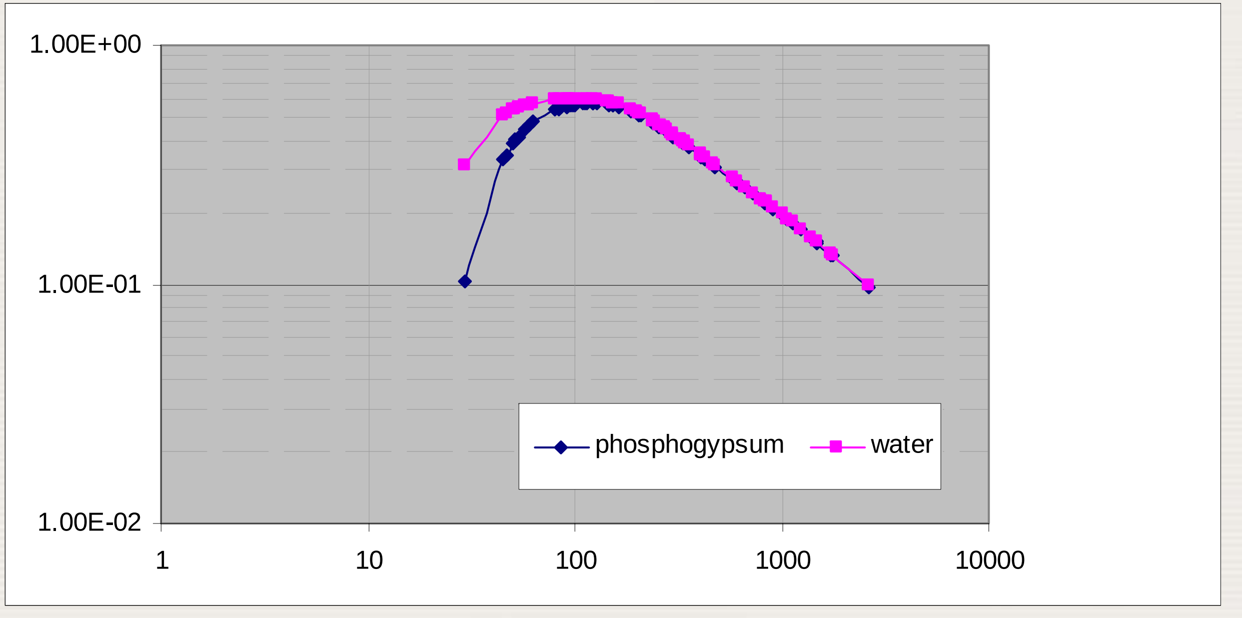
Self-attenuation: important at low energies



Well-type detector, 200% relative efficiency
Geometry: cylindrical tube r = 1cm, h = 4.5cm
Phosphogypsum density 1.26g·cm^{-3}

(Monte-Carlo calculations, GESPECOR)

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Coincidence-summing corrections: can be very important

Det 11 (50% well)
Sediment 1.3 g/cm**3

Nuclide	Energy (keV)	True coinc correction
TH-234	63	1.000
TH-234	92.8	1.000
PA-234M	1001.03	1.029
RA-226	186	
PB-214	53.23	0.338
PB-214	242	0.942
PB-214	295.22	1.024
PB-214	351.93	0.995
BI-214	609.32	0.548
BI-214	1120.29	0.465
BI-214	1238.11	0.481
BI-214	1377.67	1.267
BI-214	1729.64	2.397
BI-214	1764.54	1.023
PB-210	46.5	

Det 11 (50% well)
Sediment 1.3 g/cm**3

Nuclide	Energy (keV)	True coinc correction
AC-228	129.06	0.368
AC-228	153.98	0.516
AC-228	328	0.336
AC-228	338.32	0.888
AC-228	911.2	0.880
PB-212	238.63	0.999
BI-212	727.33	0.843
TL-208	583.19	0.496
TL-208	2614.51	0.371
BI-207	1063.66	0.468
U-235	143.76	0.961
U-235	163.33	0.890
U-235	185.71	0.961
CO-60	1173.23	0.594

Uncertainties

The uncertainty associated with calibration contributes to the final uncertainty budget of the analysis result for the sample

- Uncertainty from calibration
  - Standardized solution
  - Preparation of calibration standard
  - Counting of calibration standard
- Sample treatment and preparation for counting
- Sample counting (ISO 11929)

GUM: Guide to the Expression of Uncertainty in Measurement

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How to obtain a calibration curve? Demo: GammaVision